Development, manufacture and application of Beta-Cypermethrin aqueous capsule suspension

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Abstract: The optimal formula of Beta-Cypermethrin aqueous capsule suspension was screened out through the experiment, preparation and manufacture of basic formulas, which has no sedimentation, suspended drops of oil, peels and agglomerating and the diameter of capsule is in range of 10-30 μm, conformed to the technical requirement. The aftereffect of the 3.3% Beta-Cypermethrin aqueous capsule suspension which was manufactured according to the optimal formula was up more than 25 days. The experiments on controlling the larvae of *Dendrolinmus superans* Butler and the adults of *Xylotrechus rusticus* L were carried out with different concentrations of this chemical. The death rate reached 80% when 250 times solution of the chemical was sprayed on stem to control the larvae of *D. superans*. For control of the adults of *X. rusticus,* 200, 400 and 600 times solution of the chemical were applied and their control effects (death rate) reached 85.23%, 74.21% and 66.59% respectively. Two kinds of solution (200 times and 300 times) of the chemical were used to control the larvae of *D. superans* in large area, and the control effect for both concentrations was over 90%.

Key words: Aqueous capsule suspension; Cypermethrin-capsule, Pest control, *Dendrolinmus superans*, *Xylotrechus rusticus*.
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Introduction

Micro-capsule is a new technique for the process of chemicals formulation in recent years. It is to pack the effective component of chemicals into capsules via special processing methods and the release could be controlled (Xin *et al.* 1994; Lu and Sha 1998; Zhao 2000; Gao 2000). Micro-capsule technique can prolong the control effect, of object pest, mitigate the toxicity and harm to other animals and plants, stabilize the performance of crude chemicals, increase the resistance to UV radiation, reduce the lose caused by volatilization, and shield smell (Yuan and Huang 2000; Yuan *et al.* 2002; Zhu *et al.* 2002).

In recent years, great attentions have been drawn on the protection of environment during the control of forest pests. It is required that the pesticides used must have high efficiency, low pollution and toxicity to the environment, and little or none harm to non-destinations. In addition, for the difficulties of pesticide application in forest, it demands the chemicals with longer aftereffect to control the pest with long emergence period or un-uniform developmental stage. Many studies have been carried out on this aspect (Pan 1995; Huang and Pan 1997; Li *et al.* 1999; Tian *et al.* 2000; Han *et al.* 2001) and one of the most attractive techniques is micro-capsule technique.

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Materials and methods

Materials

Chemicals and reagents: Original powder of Cypermethrin, dimethylbenzene, dimethylformylamide, emulsifying agent 500, glutin, castor oil, Azone synergist, catalyzer b-12, catalyzer b-14, anti-corrosion agent, curing agent 112.

Instruments and vessels: Water bath pan, galvanothermy cover, electrical driving blender, electrical balance, ordinary balance, microscope, funnel, beaker, graduate, flask.

Equipment for Manufacture: Three 1000-L high pressure kettles with nip (No.1, No.2, No.3), one 3000-L high pressure kettles with nip (No.4), 500-L measure pot with staff guage, 6000-L liquid stock pot, filtering machine, magnetic force auto sucking pump, water line for liquid bottling, heat supply system, water supply system.

Study methods

Basic formula experiment

Step 1: Water was poured into water-bath pan and heated to 30 °C, then glutin was added and stirred with electrical driving blender for 2 h at 30°C until the liquid becomes transparent (Solution A).

Step 2: The quantified original powder of Cypermethrin was put into beaker, dimethylbenzene solvent and emulsifying agent as formulas were added, and then heated with water bath pan until it dissolve completely (Solution B).

Step 3: Mix Azone synergist with dimethylformylamide, catalyzer b-12 and catalyzer b-14 thoroughly to dissolve (Solution C).

Step 4: Fixed quantity of water was poured into flask and heated with galvanothermy cover to 40 °C; with stirring by electrical driving blender at 50 r/min, Solution B was gradually added and mixed for 8 min, then Solution C was added and mixed for 10 min. After then, the rotary speed of the blender was adjusted to 100 r/min and hold, and Solution A was rapidly added. This rotary speed was maintained for 10 min, then decreased to 80 r/min for 8min, 70 r/min for 8 min, 60 r/min for 10 min, and 50 r/min for 10min, after then stop stirring, placing for 12 h.

Step 5: Start the blender again to 50 r/min and hold. Curing agent 112 and anti-corrosion agent were slowly added and mixed for 8 min, then stop and keep stewing for 12 h and the end product was manufactured.

Step 6: Observe by sight whether there are sedimentations, drops of suspended oil or peels. Dilute to 100 times, 200 times and 300 times solution to observe its emulsification property and stability. Observe the formation and size of capsule by using microscope.

Process design for manufacture of the product

The equipment above may consist of a process flow capable of 2000 kg per year. The flow is as follows:

Add 600 L water into reaction kettle 1, heat to 30°C, start the blender and maintain at 50 r/min, then add a proper volume of glutin and mix at 30 °C until the solution is transparent (Solution A).

Put some original powder of Cypermethrin into reaction kettle 2 and add demethylbenzene and emulsifying agent though measure pot, then heat and stir until to completely dissolve (Solution B).

Add Azone synergist, dimethylformylamide, castor oil, catalyzer b-12 and catalyzer b-14 into reaction kettle 3, mix thoroughly to dissolve (Solution C).

Add some water into reaction kettle 4, heat to 40°C and hold, start the blender to 50 r/min and hold, add Solution B slowly and mix for 8 min, after then add Solution C and mix for 10 min. Adjust the rotate speed of the blender to 100 r/min and hold, and add Solution A rapidly. Maintain this speed for 10 min, and then decrease to 80 r/min for 8 min, 70 r/min 8 min, 60 r/min 10 min, 50 r/min 10 min. Now stop stirring, put this solution into storage pot 1 and keep quietly for 12 h (semi-manufactured goods).

Start filtering machine, and put the semi-manufactured goods in storage pot 1 into reaction kettle 4 through filtering machine. Start the blender and hold at 50 r/min, add curing agent 112 and anti-corrosion agent slowly, mix for 8min and stop. Put this solution into storage pot 2 and keep quietly for 12 h. Check the quality of this solution and pack up the qualified solution by packing machine. Now the end product is manufactured.

Results

Selection of formulas

The quality of capsule husk wrapping, size of granule, ability to diffuse and suspend, and the thickness of husk all have influence on the property and application effect of capsule products, and these indexes are directly related to the water, proportions of all kinds of stuffs and the stirring speed. Therefore, before large scale of manufacture, basic prescriptions should be tested in the laboratory.

Five formulas were tested in the laboratory (Table 1). For the qualified products, it must be stable ivory-white liquid without sedimentation, suspended drops of oil, peels and agglomerates. After diluted by water, it is also stable, without suspended oil and delamination phenomenon. Granules must be uniform and diameter is in range 10-30 μm. Individual separation of the capsules must be good and no agglomerates appear.

Talbe 1. The test result of five formulas of Beta-Cypermethrin aqueous capsule suspension

Components	Content /%				
	Formula 1	Formula 2	Formula 3	Formula 4	Formula 5
Original powder of Cypermethrin	3.3	3.3	3.3	3.3	3.3
Azone synergist	1.1	1.1	1.1	1.1	1.1
Dimethylbenzene	15	13.5	12	12	12
Dimethylformylamide	5	7	6	5.5	5.5
Catalyzer b-12	2.5	1	1.5	2	2
Catalyzer b-14	1	2.5	1.5	2	2.5
Emulsifying agent 500	3.5	1	1.5	2	2.5
Glutin	1	3	2	1.5	1.8
Castor oil	10	5	5	5.5	5.5
Anti-corrosion agent	0.3	0.3	0.3	0.3	0.3
Curing agent112	0.3	0.3	0.3	0.3	0.3
Water	57	62	65.5	64.5	63.2
Results	disqualification	disqualification	disqualification	close to grade	up to grade

For formulas 1, stable ivory-white liquid were not formed, many agglomerates or floccule occurred, and it could not dissolve when diluting with water. There was a thick oil layer on upper layer in static state. This phenomenon might be resulted from the surplus of emulsifying agent and solvent and the shortage of glutin.

The result of Formula 2 was similar to Formula 1, but there were no suspended oil on it. Microscope observation

found that regular circle capsules were formed, but the size was bigger, husk of capsule was thicker, the core was smaller, and agglomerates appeared due to the incorrect proportion of catalyzer. Surplus glutin and shortage of emulsifying agent might lead to bigger capsules and thicker husk.

For Formula 3, ivory-white liquid was formed and no agglomerates appeared. However, hard peel-like layer occurred on the surface, which did not dissolve when stirring. This might be resulted from the shortage of caster oil and catalyzer.

The product of Formula 4 approached the standard products. Only the size of capsules was inhomogeneous. Most of them were bigger than the standard one and had thinner husks. A few agglomerates still appeared. This might be also resulted from a little shortage of emulsifying agent, glutin and catalyzer b-14.

The product of formula 5 attained qualified product. The Ivory-white liquid was stable, without precipitation, suspended drops of oil, peels and agglomerates. Capsules with right size of which diameter was 10~30 μm were homogeneous.

Test of technical indexes

Beta-Cypermethrin aqueous capsule suspension is mainly used to control the pests that get down from trees for overwintering and return to trees in spring, such as the adults of longhorn beetles and the larvae of *Dendrolinmus superans* As the emergence of these pests is not at the same time, the time for pests to get dawn or climb up trees is also different, as a result, it is difficult to control the pest for a longer time through one times application of ordinarily prepared chemicals. Therefore it is needed to extend the aftereffect of chemicals as possible. The Beta-Cypermethrin aqueous capsule suspension, under the protection of husk, is hardly impacted by the environment. When pests crawl and touch capsules, capsules can kill the pest by bursting and releasing high concentration chemicals. We took edible glutin as husk, which was degradable and has no negative effects on environment. However, as the edible glutin has osmosis, the aftereffect of chemicals may be affected by sunlight, wind and blowing dust, etc. under natural condition. Therefore, the aftereffect of chemicals should be tested under natural condition in advance.

Experiment on controlling *Dendrolinmus superans*

The experiment spot was located in Pingshan Forest Farm of Acheng Forestry Bureau. This spot was within a 10-year-old pure larch stand with a canopy density of 0.7. The experiment time was from April 15 to May 5. The object insect were 2nd-instar larvae of *Dendrolinmus superans,* which were collected in its dormancy stage before moved to trees and raised in artificial climate chamber. Ten trees on different positions were selected and 250 times solution of Beta-Cypermethrin aqueous capsule suspension was sprayed on the stem of tree at the height of 1.2 m. The spray circle was 25 cm in width. Five trees as control were sprayed with water. A "V" shape plastic bag and a plastic circle were placed below and above the chemicals circle respectively. Ten larvae per tree were placed in the plastic bag every 4 days after spraying. Let the larvae crawl through the chemical circle and stop at plastic circle. The larvae which crawled though the chemical cirlcle were collected and raised, and the mortality of larvae was recorded (Table 2).

Table 2. Investigations of chemical effect for controlling the larva of *Dendrolinmus superans* by 250 times solution of 3.3% Beta-Cypermethrin Aqueous Capsule Suspension

Sample	5th day		8th day		11th day		14th day		17th day		20th days	
trees	Number	Death	number	Death	number	Death	number	Death	number	Death	number	Death
No	of Larva	rate (%)	of larva	rate (%)	of larva	rate (%)	of larva	rate (%)	of larva	rate (%)	of larva	rate (%)
1	10	100	10	100	10	100	10	100	10	100	9	90
2	10	100	10	100	10	100	10	100	9	90	8	80
3	10	100	10	100	10	100	10	100	10	100	9	90
4	10	100	10	100	10	100	10	100	9	90	8	80
5	10	100	10	100	10	100	10	100	8	80	7	70
6	10	100	10	100	10	100	10	100	9	90	9	90
7	10	100	10	100	9	90	8	80	8	80	7	70
8	10	100	10	100	9	90	9	90	8	80	8	80
9	10	100	10	100	9	90	8	80	7	70	7	70
10	10	100	10	100	10	100	9	90	9	90	8	80
ck		0		0		0		0		0		0

The experimental results (Table 2) showed that the larvae of *Dendrolinmus superans* would be killed as long as they crawled through the chemicals circle. Furthermore, the average death rate of larvae could be over 80% on the 20th day after spraying. The killing effect was very significant within 10 days after spraying. During this period the experimental larvae had a rapid dead speed, and some larvae were killed completely within only a few hours. With the increase of days between spray and insect placing, although the mortality was still high, the killing speed de-

creased greatly. The larvae could live as long as 2-3 days after touching chemicals, after then they were killed completely. In addition, the aftereffect of chemicals of the sample tree 5, 7 and 9 near the road was decreased faster, which suggested that the aftereffect of chemicals decreased with time and is also affected by wind and flowing dust. To sum up, the application of Beta-Cypermethrin aqueous capsule suspension within stands, with an appropriate higher dose, can control the larvae of *Dendrolinmus superans* in the period that the larvae moved to trees from the overwinterring place.

Experiment on controlling *Xylotrechus rusticus* L.

The application of 3.3% Beta-Cypermethrin aqueous capsule suspension for controlling *Xylotrechus rusticus* was carried out on May 25, 2003 in the agricultural shelter forest in Datong district, Daqing City. The trees were ten years old poplar, with an average tree height of 10 m, and a plant spacing of 2 m. The Beta-Cypermethrin aqueous capsule suspension was diluted to 200 times, 400 times and 600 times solution for application, and 4.5% Beta-cypermethrin emulsifiable concentrate was taken as contrast and diluted to 800 times solution. Water spaying was taken as control. All small experimental plots were randomly arranged. Four trees in each plot were sprayed, and four repeats were done for each treatment.

The experiment time was just in the period when *Xylotrechus rusticus* starts eclosion. The population density of larva on standard trees was 150-250 larvae per tree. In the whole stage of eclosion, it was estimated that 40-60 adult insects may emerge at an individual tree annually. The day of chemical spray was sunny, no rain after the next ten days. High pressure spraying head of Litter sprayer was adopted to spray chemicals, and spraying quantum for each tree was 20-25 kg.

Suitable plastic film was laid under the crown of each treatment tree to calculate the number of dead insect. The number of dead adults was investigated at the 10th h, 24th h, 3rd day, 5th day, 10th day, 15th day, and 20th day after spraying. Live insects were investigated by shaking tree. The investigations are shown in Table 3.

Table 3. The result for controlling the adults of *Xylotrechus rusticus* by spaying 3.3% Beta-Cypermethrin aqueous capsule suspension

Treatment	Times of dilution	The number of eclosion larvae	The death rate (%)					Difference in significance 0.5
			I	II	III	IV	average	
Capsule dose	200	323	83.91	87.67	84.81	84.52	85.23	a
Capsule dose	400	313	74.07	75.00	73.33	74.44	74.21	b
Capsule dose	600	305	69.88	66.18	60.00	66.28	66.59	c
Emulsifiable concentrate	800	286	28.95	31.88	32.35	30.83	30.00	d
control		56	—	—	—	—	3.57	

Investigation result shows that 3.3% Beta-Cypermethrin aqueous capsule suspension has good result on controlling the adults of *Xylotrechus rusticus*. The pest could be killed as soon as 1-2 h after spraying. The caliberated death rates were 84.68%, 73.49% and 65.03% for application of 200 times, 400 times and 600 times solution respectively. The amount of eggs of *Xylotrechus rusticus* also decreased sharply, for the chemical can kill the adults directly, thus the damage of larvae were greatly reduced.

Controlling *Dendrolinmus superans* in large area with Beta-Cypermethrin aqueous capsule suspension

In the spring of 2003, we used 3.3% Beta-Cypermethrin aqueous capsule suspension to control *Dendrolinmus superans* in large area at the Hongqi Forest Farm of Hegang Municipal Forest Bureau and the Green Forest Farm of Gannan County, Heilongjiang Province. Hongqi Forest Farm is in mountainous areas. Its woodland is 15-year-old pure larch, with a canopy density of more than 0.8, and undergrowth is very thick. Green Forest Farm is flat, and forest is 20-year-old artificial pure larch, with the canopy density of 0.6-0.8.

Spraying was carried out before the larvae of *Dendrolinmus superans* crawl up the trees, on May 20, 2003 in Hegang and on May 25, 2003 in Gannan. The larvae were 3-4-instar larvae. The aqueous capsule suspension was diluted to 200 times and 300 times solutions by clear water. The solutions were sprayed on the tree to form a close poisonous circle with the width of 25 cm at trunk height of 1.2 m. Three repeats were made for each kind of chemical solution used in each of three separate forest stands. The area of each stand is at least 333 m^2. Five trees were selected as sampling investigation in each treatment area. Vegetation under the projection of tree crown was cleared away in order to investigate larvae falling to the ground. Since the temperature was relatively low in early spring, no natural enemy or microbe could affect the death rate of larvae, so contrast treatment was not done.

The control effect was investigated at 8:00 a.m. and 16:00 p.m. every day after the chemical was sprayed for 24 h. The larvae that falled from tree to the ground were counted. All the live larvae were put into the feeding coop, and dead ones were thrown away. The number of dead larvae, death speed and death condition were recorded. The investigation was carried out until the 25th day when *Dendrolinmus superans* stopped going up the tree. We counted numbers of all dead larvae and live ones in the feeding coops, and we also investigated the number of live

larvae on the tree by shaking the tree. The total number of living larvae include those on the sample trees and in the feeding coop. The investigating results were shown in Table 4.

Table 4 The control effect of *D. superans* larvae by spraying 3.3% Beta-Cypermethrin aqueous capsule suspension

Chemical solution /times	place	re-peat	Total number of larvae	Death number (head)						Number of live larvae	Control effect %	Average effect of control /%
				24 h	120 h	240 h	360 h	480 h	600 h			
200	Gannan	1	1785	80	180	445	585	330	110	55	96.9	97.5
		2	1523	59	151	406	505	296	79	27	98.2	
		3	1664	73	162	426	557	314	89	43	97.4	
	Hegang	1	1213	41	111	395	430	170	45	21	98.3	98.6
		2	743	29	64	233	299	87	21	10	98.7	
		3	531	17	37	145	256	58	12	6	98.9	
300	Gannan	1	1842	84	179	343	527	281	254	174	90.6	90.5
		2	1518	54	161	305	419	204	226	149	90.2	
		3	1767	69	158	359	464	297	256	164	90.7	
	Hegang	1	1213	37	74	298	387	159	256	94	92.3	92.6
		2	728	31	67	207	216	56	165	58	93.6	
		3	625	19	42	137	196	32	147	45	92.8	

Note: every investigation number is the total larva numbers from five sample trees under the same treatment in five days.

Conclusion and discussion

The experimental results showed that 3.3% Beta-Cypermethrin aqueous capsule suspension has better force to kill *Dendrolinmus superans*, especially during the early ten days of applying chemicals. The experiment worm to be poisoned die in the period from 4th hour to 24th hour, but as the time prolongs the efficacy of the chemical began to reduce, the poisoned pest die completely after living 2 or 3 days. The mortality of *D. superans* was over 97% when 200 times chemical solution was sprayed, and the larvae to be poisoned died quickly. The control effect of 300 times chemical solution is close to that of 200 times solution in the early ten days, but as the time prolonged, the death speed of larvae decreased obviously, and some larvae live for several days before die thoroughly. Many experimental larvae falled from trees on 25th day, some of them were poisoned but did not die, so the final control effect (90%-92%) was lower than that of 200 times solution..

The application time and dose of the chemical should be accurately determined when using Beta-Cypermethrin aqueous capsule suspension to control *D. superans*. Good control result can be obtained by spraying the chemical on proper height of trees during the period when the larvae of *D. superans* are crawling up trees.

This aqueous capsule suspension has special and applicable value in forest, especially in the middle age and sparse forest that are tall and thick. Although the chemical has better effect in control of *Xylotrechus rusticus* L, the validity period is not long enough, as a result, the capsule rind should be regulated further to make the aftereffect to be as long as 30-40 days.

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